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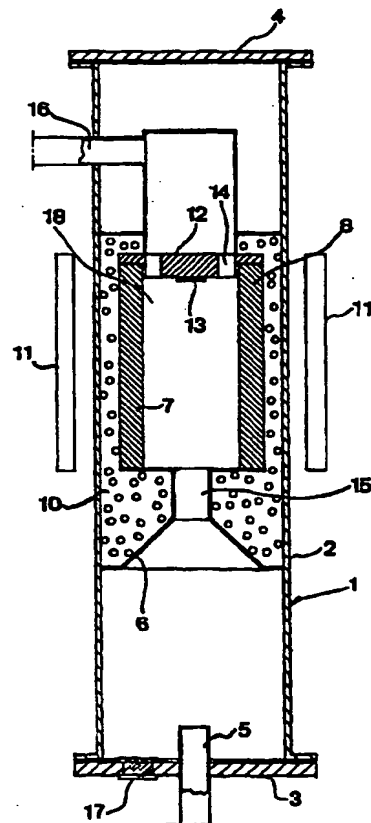
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(54) Title: A DEVICE AND A METHOD FOR EPITAXIALLY GROWING OBJECTS BY CVD

(57) Abstract

A device for epitaxially growing objects of SiC, a group III-nitride or alloys thereof by Chemical Vapour Deposition on a substrate (13) comprises a susceptor (7) having circumferential walls (8) surrounding a room (18) for receiving a substrate and means (11) for heating said circumferential susceptor walls and by that the substrate and a gas mixture fed to the substrate for the growth by feeding means (5). The heating means (11) is arranged to heat the susceptor (7) and by that the substrate (13, 13') above a temperature level from which sublimation of the material grown starts to increase considerably, and the feeding means is arranged to feed said gas mixture with such a composition and at such a rate into the susceptor that a positive growth takes place.



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A device and a method for epitaxially growing objects by CVD

TECHNICAL FIELD OF THE INVENTION AND PRIOR ART

- 10 The present invention relates to a device for epitaxially growing objects of SiC, a group III-nitride or alloys thereof by Chemical Vapour Deposition on a substrate comprising a susceptor having a circumferential wall surrounding a room for receiving the substrate and means for heating said circumferential susceptor walls and thereby the
- 15 substrate and a gas mixture fed to the substrate for the growth by feeding means as well as a method for epitaxially growing such objects by Chemical Vapour Deposition on a substrate according to the preamble of the first appended independent method claim.
- 20 Accordingly, the invention is applicable to the growth of SiC, group III-nitrides and all types of alloys thereof, but the common problem of growing such objects of a high crystalline quality and at a reasonable grow rate from the commercial point of view will now by way of a non-limitative example be further explained for SiC.
- 25 SiC single crystals are in particular grown for being used in different types of semiconductor devices, such as for examples different types of diodes, transistors and thyristors, which are intended for applications in which it is possible to benefit from the superior properties of
- 30 SiC in comparison with especially Si, namely the capability of SiC to function well under extreme conditions. The large band gap between the valence band and the conduction band of SiC makes devices fabricated from said material able to operate at high temperatures, namely up to 1000 K.
- 35 However, high temperatures are needed for obtaining a good ordered growth thereof. The epitaxial growth of silicon carbide by Chemical

Vapour Disposition is therefor carried out in a temperature regime of 1400-1700°C. These high temperatures are needed both to obtain decomposition by cracking of the Si- and C-containing precursor gases of said gas mixture and to ensure that the atoms are deposited on the substrate surface in an ordered manner. However, high temperatures also mean problems with impurities coming out of different types of material, so that the temperature could until now not be raised above said temperature interval, which results in such a low grow rate (some μm per hour) that it is out of the question to grow boules for forming i.a. substrates by using CVD, so that this method is only used for growing objects in the form of layers. However, it is not possible to even grow layers of SiC by CVD through the devices already known at such a high grow rate that a commercial production thereof will be really interesting. A raise of the temperature has not been tried, since that would have resulted in a rapid degradation of the walls of the susceptor due to an increased etching of hot spots thereof leading to an unacceptable incorporation of impurities therefrom into the layers grown. It is also assumed that crystalline imperfections reduces the minority carrier lifetime. A high crystalline perfection may be obtained at high temperatures or at low grow rates. The minority carrier lifetime governs the forward conduction losses and switching losses of a power device. The minority carrier lifetime is also reduced by the introduction of unwanted compensating acceptors in the N-type layer grown, so that a minority carrier lifetime acceptable for high-power bipolar devices could not be obtained until now. It is therefore important to produce layers of a high crystalline perfection at high grow rates without the introduction of compensating acceptors. Common impurities as nitrogen and oxygen may also be incorporated at lower concentrations at higher growth rates which also may influence the minority carrier lifetime positively.

As already mentioned, it is due to said low grow rates impossible to grow boules, which require grow rates in the order of millimetres per hour, by CVD, so that the seeded sublimation technique is presently used for growing boules, which may then be sliced to substrates. However, the crystalline quality of the boules grown by this technique is low in comparison with that of the SiC layers epitaxially grown by

CVD. The substrates produced in this way are perforated by small holes called micropipes or pinholes, which limit the device area considerably which consequently make high-power devices of SiC not commercially interesting yet. In the seeded sublimation technique the source is a SiC powder that sublimates, whereupon the gas species are transported by a temperature gradient to the seed crystal where the growth occurs. The conditions in the gas phase are governed by thermodynamics only, which makes it difficult to keep the C/Si ratio constant due to Si vapour leakage out of the system. Furthermore, the purity of the gases used for Chemical Vapour Deposition are several orders of magnitude higher than that of the source material used for seeded sublimation growth.

SUMMARY OF THE INVENTION

The object of the present invention is to advise a solution to the problems discussed above by providing a device and a method making it possible to epitaxially grow objects by Chemical Vapour Deposition at a high grow rate while still obtaining a high crystalline quality of the object grown.

This object is in accordance with the invention obtained by arranging said heating means to heat the susceptor and by that the substrate above the temperature level from which sublimation of the material grown starts to increase considerably, and by arranging said feeding means to lead said gas mixture with such a composition and at such a rate into the susceptor that a positive growth takes place. It has surprisingly been found that a raise of the temperature above said temperature level makes it possible to obtain a considerably increased grow rate in comparison to Chemical Vapour Deposition carried out at normal temperatures while still achieving a high quality of the object grown. Accordingly, the temperature is raised to a temperature normally used for growing boules by the seeded sublimation technique. A method according to the invention which may be carried out by using the device according to the invention is defined in the first appended independent method claim.

In the SiC case the grow rates may be increased by 2-3 orders of magnitude compared to the conventional CVD technique, so that high quality layers may be produced at rates being commercially interesting. Furthermore, the etching by hydrogen or other etching gases of the object grown on said substrate is increased considerably at these higher temperatures, and it has been found that polycrystalline regions, i.e. regions with a lower quality, are more rapidly etched than monocrystalline regions, i.e. regions with a high quality, which results in a higher quality of the crystal grown. Furthermore, it has been found that the concentration of unwanted charge carriers will be dramatically reduced when the grow rate is increased, this effect being rather exponential than proportional, which will make it possible to obtain prolonged minority carrier lifetimes, so that it will be possible to reduce the introduction of unwanted compensating acceptors into SiC layers grown and thereby high-power bipolar devices with a sufficiently long minority carrier lifetime may be produced.

It has also surprisingly been found that the grow rates obtained by the temperature increase according to the invention are high enough for making it very interesting to grow boules by the CVD technique. Thus, it is possible to grow boules while using a gas mixture normally used in the CVD technique, which means a purity of the components used for said growth of several orders of magnitude higher than the source material used for seeded sublimation growth. Furthermore, it will be possible to control the growth process by varying the fluxes of the precursor gases independently, which makes it possible to hold the C/Si-ratio constant during the growth. Additionally, the degree of supersaturation which influences both the grow rate and the formation of micropipes can be varied without any thermal gradient. Thus, boules having a superior crystalline quality to that obtained by using the seeded sublimation technique may be obtained at commercially interesting grow rates through the High Temperature Chemical Vapour Deposition (HTCVD) according to the invention.

According to a preferred embodiment of the invention said circumferential susceptor walls have a substantially uniform thickness. This will make it possible to raise the temperature above said temperature

level without any further measures in comparison to already known hot-wall susceptors, because no hot spots, i.e. concentrations of heat, in the susceptor walls with an exaggerated etching as a result will be created, so that the temperature for the entire susceptor may be raised to the level and above which said hot spots had in the susceptors according to the prior art without giving rise to additional problems with incorporation of impurities from said walls into the object grown.

According to another preferred embodiment of the invention said circumferential walls of the device form a substantially cylindrical susceptor. It has turned out that this shape of the susceptor will be particularly advantageous for easily obtaining a uniform temperature over the entire susceptor wall while avoiding hot spots.

According to still another preferred embodiment of the invention the inner walls of the susceptor are made of or covered by a plate made of SiC, an alloy of SiC and the material grown or the material grown. Such a plate will contribute to prevent impurities from the walls of the susceptor from being set free and being incorporated in the object grown.

The invention also relates to a method according to the second independent method claim, which in accordance with the invention is characterized in that the etching action of said gas mixture upon the susceptor and substrate is varied by varying the content of at least one etching gas in said gas mixture. It is known that the etching action is increasing with the temperature, and it will increase considerably above a certain temperature, which is the temperature normally used for growing said objects by Chemical Vapour Deposition. It is desired to have comparatively extensive etching of the substrate or more exactly the object grown thereon during the growth for removal of polycrystalline regions formed during the growth, but the temperature dependence of the etching action will normally mean that the etching action is too low at low temperatures and too high at high temperatures. Thanks to the inventional characteristic of varying the contents of at least one etching gas in said gas mixture the etching action may be held at a desired level for different temperatures. Accordingly, it

will be possible to raise the etching action at lower temperatures by increasing the content of an etching gas in the gas mixture and the etching action may be lowered at high temperatures by reducing the content of said etching gas at these temperatures or alternatively
5 varying the content of a non-etching gas in said gas mixture. This will make it possible to grow layers and boules by Chemical Vapour Deposition at high temperatures while holding the etching action of the gas mixture at an optimal level. Accordingly, the etching may be held below a critical value also at very high temperatures, so that the
10 etching of the susceptor walls may be held at the same level as in the growth by CVD at the temperatures according to the prior art resulting in the possibility of obtaining high grow rates. This characteristic may very preferably be combined with the characteristic of using a susceptor having circumferential walls of a substantially uniform thickness.

15 According to still a further preferred embodiment of the invention the substrate is heated by heating the susceptor above a temperature level from which sublimation of the material grown starts to increase considerably, and the content of said at least one etching gas in said
20 gas mixture and the supply rate of precursor gases included in the gas mixture for said growth are regulated so that a positive growth takes place, i.e. the deposition rate of elements forming the material grown on the substrate is higher than the rate of material leaving the layers on the substrate due to sublimation and etching. This controlled
25 interaction between deposition on one hand and sublimation and etching on the other taking place at high temperatures, which in the SiC case means temperatures within the temperature range used when boules of SiC are produced by the conventional seeded sublimation technique, leads to a high crystalline quality of the object grown while
30 growing it at high grow rates. Thus, the comparatively extensive etching and sublimation during the growth will counteract the formation of crystal defects and the incorporation of unwanted impurities, such as unwanted compensating acceptors, into the object grown.

35 Further preferred features and advantages of the device and method according to the invention will appear from the following description and the other dependent claims.

BRIEF DESCRIPTION OF THE DRAWINGS

With reference to the appended drawings, below follows a specific description of a preferred embodiment of the invention cited as an example.

In the drawings:

Fig 1 is a longitudinal cross-section view of a device according to a first embodiment of the invention,

Fig 2 is a perspective view of the susceptor used in the device of Fig 1 according to a first preferred embodiment adapted for epitaxially growing layers, parts of the susceptor wall being broken away so as to illustrate the interior of the susceptor, and

Fig 3 is a view similar to Fig 2 of a susceptor according to a second preferred embodiment adapted for growing boules by Chemical Vapour Deposition.

DETAILED DESCRIPTION OF PREFERRED EMBODIMENTS OF THE INVENTION

Fig 1 shows schematically a device according to a preferred embodiment of the invention for epitaxially growing SiC by Chemical Vapour Deposition on a SiC-substrate in a simplified manner, and it is obvious that the device in question also comprises other means, such as pumps, but conventional equipment having nothing to do with the invention has been omitted for the sake of clearness and concentration to the inventional characteristics. The device comprises a vacuum casing 1 constituted by a tube 2 of quartz extending substantially vertically and two opposite end flanges 3 and 4. The end flange 4 is preferably removable so as to get access to the interior of the tube 2. A conduit 5 for supplying a stream of a gas mixture intended for the growth of a crystal is introduced through the lower end flange 3. The conduit 5 is connected to separate conduits leading to sources for the

respective components of said gas mixture and these conduits are provided with flow regulating means not shown for regulating the content of each component in said gas mixture as desired.

- 5 Furthermore, the device comprises a funnel 6 for concentrating the gas flow from the conduit 5 into a susceptor 7 (see also Fig 2). The susceptor 7 shown in Figs 1 and 2 is adapted for epitaxially growing layers of SiC. The susceptor is substantially cylindrical with circumferential walls 8 of a substantially uniform thickness. The walls are made
10 of graphite, but they are internally coated by a layer of SiC 9 or alternatively covered by a cylindrical plate made of SiC. The space surrounding the susceptor is enclosed and filled by graphite foam 10 for thermal insulation for protecting the surrounding quartz tube 2. Rf-field radiating means 11 in the form of a Rf-coil surrounds the tube 2
15 along the longitudinal extension of the susceptor 7. This heating means 11 is arranged to radiate a Rf-field uniformly heating the walls 8 of the susceptor and thereby the gas mixture introduced into the susceptor.
- 20 The susceptor 7 comprises a lid 12 of the same material as the rest of the susceptor, on the lower side of which a SiC substrate 13 is arranged and which may be removed from the rest of the susceptor so as to remove the substrate after a layer has been grown thereon. The lid 12 is provided with peripheral gas outlet holes 14, so that a
25 preferable laminar gas flow will enter the susceptor room 18 through the lower inlet 15 and flow close to the substrate and leave the susceptor through the upper outlets 14 and then the device through a conduit 16 connected to a pump not shown.
- 30 The temperature inside the susceptor 7 may be checked pyrometrically through looking into the susceptor 7 through a window indicated at 17.

The function of the device is as follows: a gas mixture including Si- and C-containing precursor gases and one or more carrier gases is led
35 through the conduit 5 in the direction of the susceptor inlet 15. The Si-containing precursor gas is preferably silane, while the C-containing precursor gas is propane or eventually methane. The carrier gas may

be H₂, but there may also be a content of a non-etching carrier gas, such as Ar, for a purpose which will be explained below. The heating means 11 will heat the susceptor walls uniformly to a temperature of about 2200°C, which is possible thanks to the substantially uniform thickness thereof resulting in no so called hot spots of the susceptor walls. The precursor gases entering the susceptor 7 will be heated through the susceptor and cracked into Si- and C-atoms, which will be transported to the substrate 13 and deposited thereon for epitaxially growing layers of SiC thereon. Thanks to the vertical arrangement of the susceptor walls the flow upwards of the hot gases heated will be promoted, so that the gases will be better utilized for the growth. The high temperature will lead to a continuous sublimation of SiC from the substrate as well as a considerable etching of the substrate through the etching carrier gas component (H₂), but the supply rate of the precursor gases and the carrier gas or gases is regulated so that a positive growth takes place, i.e. the deposition rate of elements forming the SiC layers grown on the substrate is higher than the rate of material leaving the layers on the substrate due to sublimation and etching. This interaction of deposition on one hand and etching and sublimation on the other for said growth promotes the formation of a crystal with a high crystalline quality, especially since the etching of polycrystalline regions is faster than that of monocrystalline regions. However, an increase of the content of a non-etching carrier gas with a reduction of the content of an etching carrier gas in the gas mixture while raising the temperature will give rise to an etching being below a critical level even at the high temperatures used. This means that the walls of the susceptor may be spared in spite of the high temperatures prevailing. Ar may be used as non-etching carrier gas and H₂ as etching carrier gas.

The initial stage of the growth is particularly important for the quality of the layers grown also thereafter. The grow rate may therefore be kept low at the initial stage of growth to form a smooth uniform layer after which the grow rate gradually can be increased to 100 µm - several millimetres/hour depending on the temperature. Thus, SiC layers may in this way be grown at a high grow rate, possibly from 100 µm to a few millimetres per hour, on the substrate 13, and this high grow rate

with said continuous sublimation and etching will give rise to a high quality of the layer grown in spite of the high temperatures, and due to the higher crystalline quality obtained at these temperature and due to a faster healing of crystalline imperfections propagating from the substrate the introduction of unwanted compensating acceptors into the layers grown is dramatically reduced with respect to the epitaxial growth of such layers by CVD at normal temperatures resulting in a significant prolongation of the minority carrier lifetime in the crystals grown. This is a vital improvement for the production of high-power bipolar devices.

Fig 3 shows a susceptor 7' according to a second preferred embodiment of the invention, which is adapted for growing boules 19 of SiC on a substrate in the form of a seed crystal indicated at 13'. This susceptor is intended to be incorporated in a device according to Fig 1 in the same way as the susceptor according to Fig 2. The susceptor according to Fig 3 only differs from that according to Fig 2 by the arrangement of gas outlet holes 14' at the bottom of the susceptor. Accordingly, the gas streams will as indicated reach the region of the SiC boules grown where the cracked precursor gas components will be deposited and the resulting components thereof will be diverted back and leave the susceptor through the holes 14'.

SiC boules with a high crystalline quality may in this way be epitaxially grown by CVD at a sufficiently high grow rate thanks to the high temperature used. The grow rate may advantageously be higher than 1 millimeter per hour. The C/Si ratio in the susceptor may be held constant thanks to the possibility to vary the content of the precursor gases in the gas mixture. This means that the degree of supersaturation which influences both the grow rate and the formation of micropipes can be varied without any thermal gradient. Additionally, the purity of the gases used are several orders of magnitude higher than the source material used for seeded sublimation growth, so that the crystalline quality of the boules will be much higher than before. The control of the etching is carried out in the way described above for the growth of layers.

The invention is of course not in any way restricted to the preferred embodiment of the device and method described above, but several possibilities to modifications thereof would be apparent for a man skilled in the art without departing from the basic idea of the invention.

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As already mentioned the invention is also applicable to the growth of a group III-nitride, an alloy of group III-nitrides or an alloy of SiC and one or more group III-nitrides, for which the corresponding positive result may be expected.

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The definition "object" in the claims is made for including the epitaxial growth of all types of crystals, such as layers of different thicknesses as well as thick boules.

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All definitions concerning the material of course also include inevitable impurities as well as intentional doping.

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The claim definition "varying the content" is intended to also comprise a constant supply of the gas in question with a variation of other components of the gas mixture indirectly leading to a variation of the proportion of said gas in the gas mixture.

Claims

1. A device for epitaxially growing objects of SiC, a Group III-nitride or alloys thereof by Chemical Vapour Deposition on a substrate (13, 13') comprising a susceptor (7, 7') having circumferential walls (8) surrounding a room (18) for receiving the substrate and means (11) for heating said circumferential susceptor walls and by that the substrate and a gas mixture fed to the substrate for the growth by feeding means, characterized in that said heating means (11) is arranged to heat the susceptor (7) and by that the substrate (13, 13') above a temperature level from which sublimation of the material grown starts to increase considerably, and that said feeding means is arranged to feed said gas mixture with such a composition and at such a rate into the susceptor that a positive growth takes place.
2. A device according to claim 1, characterized in that said circumferential susceptor walls (8) have a substantially uniform thickness.
3. A device according to claim 2, characterized in that said circumferential walls (8) form a substantially cylindrical susceptor (7, 7').
4. A device according to any of claims 1-3, characterized in that the inner walls of the susceptor are made of or covered by a plate made of SiC, an alloy of SiC and the material grown or the material grown.
5. A device according to any of claims 1-4, characterized in that it is adapted for growing boules (19) and said substrate (13') is a seed crystal.
6. A device according to claim 5, characterized in that said heating means (11) is arranged to heat the susceptor walls (18) to a temperature within the temperature range used when such boules are produced by a conventional seeded sublimation technique.
7. A device according to any of claims 1-4, characterized in that it is adapted for growing layers.

8. A device according to any of claims 1-7, characterized in that it is adapted for growing objects of SiC, and that said heating means (11) is arranged to heat the susceptor walls (8) to a temperature above 1900°C.

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9. A device according to claim 8, characterized in that said heating means (11) is arranged to heat the susceptor walls (8) to a temperature between 2000 and 2500°C.

10 10. A device according to any of claims 1-9, characterized in that the susceptor (7, 7') is adapted to be positioned with the circumferential walls (8) extending substantially vertically in the direction of the gas feed into the susceptor.

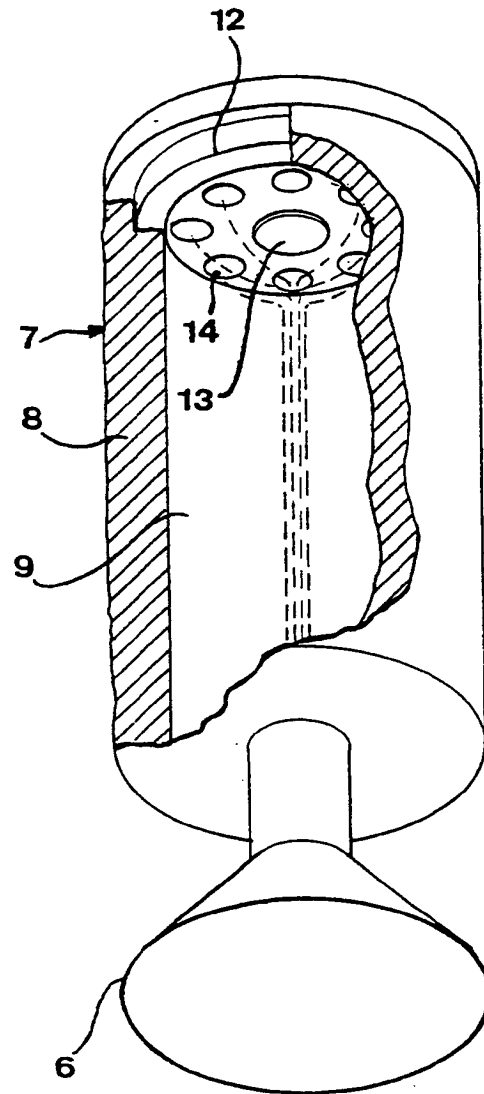
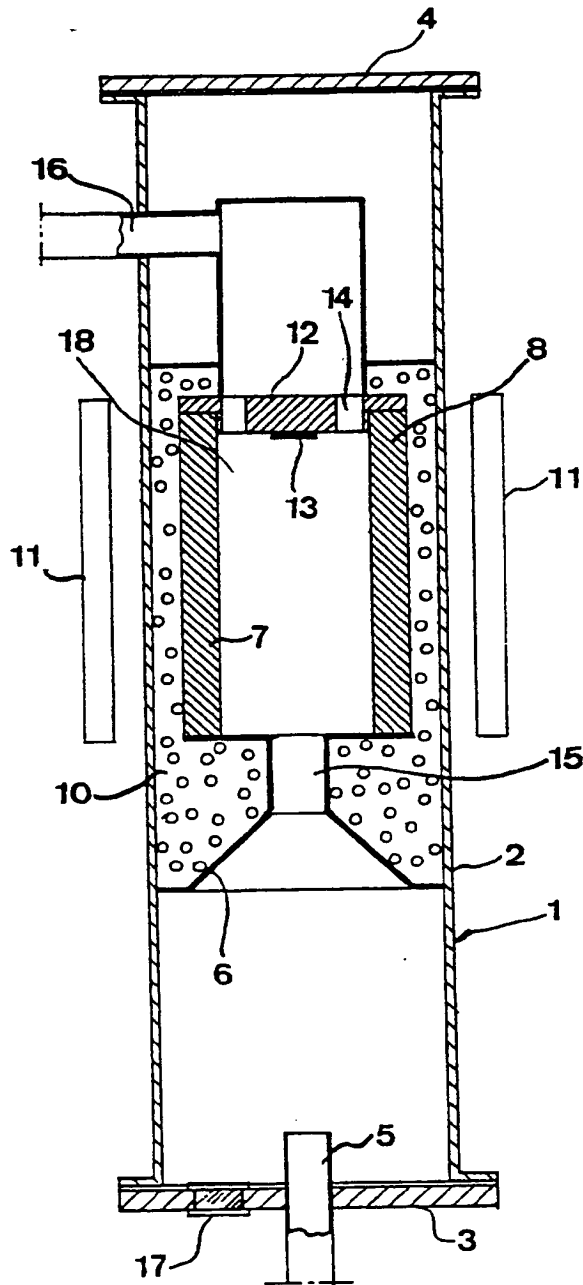
15 11. A device according to any of claims 1-10, characterized in that said heating means (11) is a Rf-field radiating means surrounding said circumferential susceptor walls (8).

20 12. A method for epitaxially growing objects of SiC, a Group III-nitride or alloys thereof by Chemical Vapour Deposition on a substrate (13, 13') received in a susceptor (7, 7') having circumferential walls, said circumferential susceptor walls and by that the substrate and a gas mixture led to the substrate for the growth being heated, characterized in that the susceptor (7) and by that the substrate (13, 13') is
25 heated above a temperature level from which sublimation of the material grown starts to increase considerably, and that said gas mixture is fed with such a composition and at such a rate into the susceptor that a positive growth takes place.

30 13. A method for epitaxially growing objects of SiC, a Group III-nitride or alloys thereof by Chemical Vapour Deposition on a substrate (13, 13') arranged to be received by a susceptor (7, 7'), said substrate and a gas mixture fed to the substrate for said growth being heated through heating of the susceptor, characterized in that the etching action of
35 said gas mixture upon the susceptor and substrate is varied by varying the content of at least one etching gas in said gas mixture.

14. A method according to claim 13, characterized in that said etching action is varied by varying the content of a carrier gas of said gas mixture.
- 5 15. A method according to claim 14, characterized in that said content is varied by increasing the content of a non-etching carrier gas with a reduction of the content of said etching carrier gas in said gas mixture and vice versa.
- 10 16. A method according to claim 15, characterized in that the content of said non-etching carrier gas is increased upon an increase of the temperature of the susceptor and by that of the gas mixture.
- 15 17. A method according to claim 15 or 16, characterized in that said non-etching gas is Ar.
18. A method according to any of claims 13-17, characterized in that said etching gas is H₂.
- 20 19. A method according to claim 13, characterized in that the substrate is heated by heating the susceptor (7, 7') above a temperature level from which sublimation of the material grown starts to increase considerably, and that the content of said at least one etching gas in said gas mixture and the supply rate of precursor gases included in
25 the gas mixture for said growth are regulated so that a positive growth takes place, i.e. the deposition rate of elements forming the material grown on the substrate is higher than the rate of material leaving the layers on the substrate due to sublimation and etching.
- 30 20. A method according to claim 19, characterized in that SiC is grown and that the substrate is heated at a temperature above 1900°C.
- 35 21. A method according to claim 20, characterized in that the substrate is heated at a temperature between 2000 and 2500°C.

22. A method according to any of claims 13-21, characterized in that boules (19) are grown on a substrate (13') in the form of a seed crystal.
- 5 23. A method according to claim 22, characterized in that said heating is carried out at a temperature within the temperature range used when such boules are produced by a conventional seeded sublimation technique.
- 10 24. A method according to any of claims 13-21, characterized in that layers are grown on the substrate (13).



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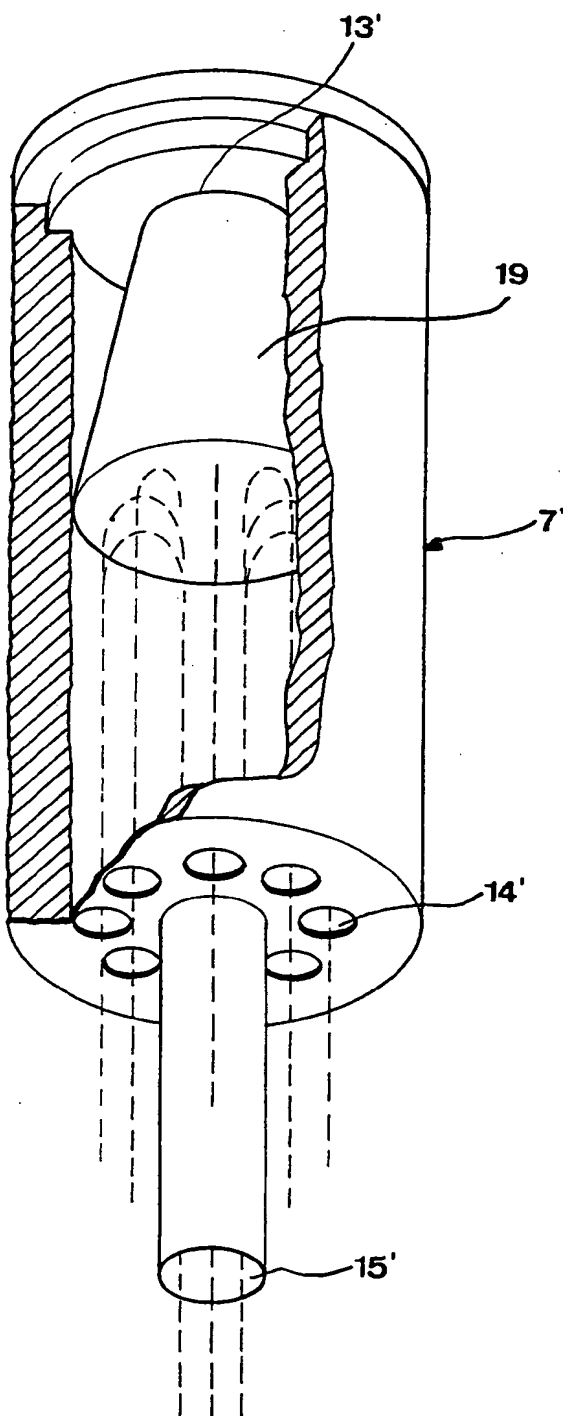


Fig 3

INTERNATIONAL SEARCH REPORT

International application No.

PCT/SE 96/00822

A. CLASSIFICATION OF SUBJECT MATTER

IPC6: C30B 29/36, C30B 29/38, C30B 23/02, C30B 25/02
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC6: C30B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

WPI, CAS

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP 0554047 A1 (NISSHIN STEEL CO., LTD.), 4 August 1993 (04.08.93) --	1-24
A	J. ELECTROCHEM.SOC., Volume 137, No 11, November 1990, D.H. KUO ET AL, "The Effect of CH ₄ on CVD beta-SiC Growth", page 3688 - page 3692, see page 368, line 31 - line 66 --	1-24
A	APPL.PHYS.LETT., Volume 66, No 11, March 1995, O. Kordina et al, "High quality 4H-SiC epitaxial layers grown by chemical vapor deposition", page 1373 - page 1375, see 1373, line 15 - line 26 -- -----	1-24

☐ Further documents are listed in the continuation of Box C.☒ See patent family annex.

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Information on patent family members

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